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Report on Recommended Specifications for Microchemical Apparatus

Volumetric Glassware. Microliter Pipets

Committee on Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society

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THE Committee on Microchemical Apparatus has published recommended specifications (1, 2) for a number of pieces of apparatus which were either the most widely used for the work in question or else an improvement over such apparatus according to tests made by members of the committee or cooperating chemists. The last report included recommended specifications for microvolumetric flasks of a new design, special measuring micropipets ranging in capacity from 0.2 to 3 ml., which increased the usefulness of these flasks, and several microcentrifuge tubes. In this report, specifications are recommended for micropipets in the microliter range.

In preparation for the work, contact was made with several interested per-

sons, both users and manufacturers, for preliminary ideas. The suggestions received were incorporated into tentative specifications and these together with a questionnaire were sent to 75 interested persons. The questions requested information regarding the types, sizes, quantities, etc., used, as well as criticisms of the tentative specifications. There were returns from 56 individuals, 48 of whom were users of this type of pipet. The information received was studied and used as a basis for changes in the specifications. Samples prepared accordingly were tested by chemists experienced in their use and proved to be satisfactory. The resulting recommended specifications for the pipet sizes most widely used are shown in Figures 1 and 2; the former are for the

capacity range of 1 to 4 μ l. and the latter for 5 to 500 μ l. The dimensions of these pipets are such that they may be used with the microvolumetric flasks for which recommended specifications were given in the last report.

For calibration of microliter pipets, the following procedure is recommended (3):

Standard procedure for micropipet calibration consists of filling the pipet with mercury, discharging the mercury into a porcelain dish, weighing the mercury, and making the appropriate weight-temperature-volume calculation. This method has been compared with that of weighing the pipet both empty and mercury-filled, and has been found less difficult and equally precise. It is

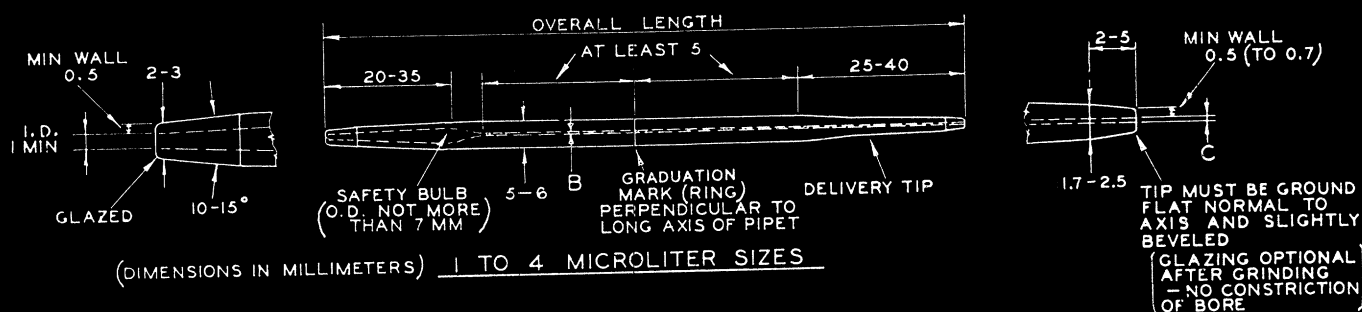


Figure 1. Microliter pipet for 1- to 4- μ l. sizes

Size, μ l. (Calibd. to Contain)	Over-all Length, Mm.	I.D. Tubing, Mm. B	I.D. at End, Mm. C	Min. Cap. Safety Bulb, μ l.	Vol. Tol., ^a %
1	140 \pm 5	0.12-0.16	0.10-0.20	50	\pm 1
2	140 \pm 5	0.16-0.25	0.15-0.25	50	\pm 1
3	140 \pm 5	0.20-0.28	0.15-0.25	50	\pm 1
4	140 \pm 5	0.24-0.32	0.15-0.25	50	\pm 1

^a Closer volumetric standardization must be carried out by user with substances under actual conditions of use.

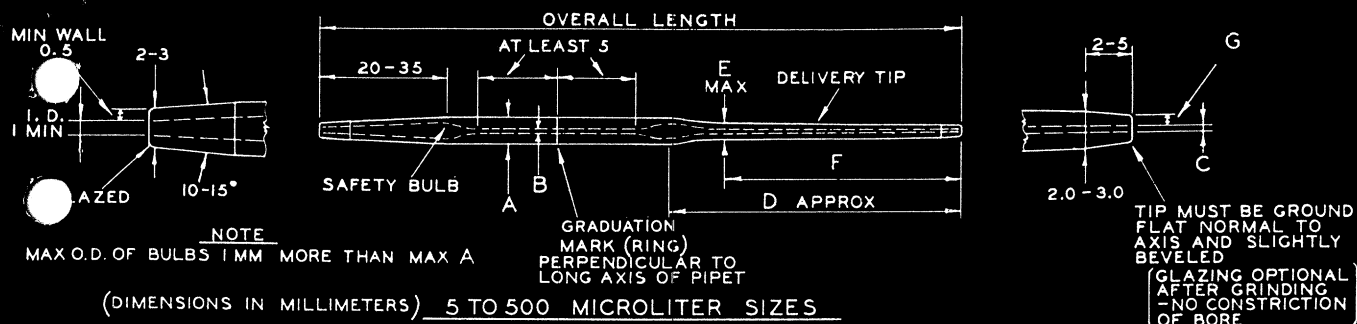


Figure 2. Microliter pipet for 5- to 500- μ l. sizes

Size, μ l. (Calibd. to Contain)	Over-all Length, Mm.	Delivery Stem							Min. Cap., Safety Bulb μ l.	Vol. Tol., %
		O.D. Tubing, Mm. A	I.D. Tubing, Mm. B	Approx. length, mm. D	Max. O.D., mm. E	Min. length, mm. F	Wall at end, Mm. G	I.D. at end, Mm. C		
5	140 \pm 5	5-6	0.18-0.25	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
6	140 \pm 5	5-6	0.18-0.25	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
7	140 \pm 5	5-6	0.18-0.25	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
8	140 \pm 5	5-6	0.18-0.25	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
9	140 \pm 5	5-6	0.18-0.25	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
10	140 \pm 5	5-6	0.20-0.35	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
15	140 \pm 5	5-6	0.25-0.40	65	4	55	0.5-0.7	0.15-0.25	50	\pm 0.5
20	140 \pm 5	5-6	0.35-0.50	65	4	55	0.5-0.7	0.25-0.50	50	\pm 0.5
25	140 \pm 5	5-6	0.35-0.50	65	4	55	0.5-0.7	0.25-0.50	50	\pm 0.5
35	140 \pm 5	5-6	0.35-0.50	65	4	55	0.5-0.7	0.25-0.50	50	\pm 0.3
50	140 \pm 5	5-6	0.35-0.50	65	4	55	0.5-0.7	0.25-0.50	50	\pm 0.3
60	140 \pm 5	5-6	0.40-0.55	65	4	55	0.5-0.7	0.30-0.50	50	\pm 0.3
75	140 \pm 5	5-6	0.40-0.60	65	4	55	0.5-0.7	0.30-0.50	75	\pm 0.3
100	140 \pm 5	5-6	0.50-0.75	65	4	55	0.5-0.7	0.30-0.50	75	\pm 0.3
150	140 \pm 5	5-6	0.75-1.00	65	4	55	0.5-0.7	0.40-0.60	100	\pm 0.3
200	145 \pm 10	5-6	0.75-1.00	65	4	55	0.6-0.8	0.40-0.60	100	\pm 0.2
250	145 \pm 10	5-6	0.75-1.00	65	4	55	0.6-0.8	0.40-0.60	100	\pm 0.2
300	145 \pm 10	5-6	0.75-1.00	65	4	55	0.6-0.8	0.40-0.70	200	\pm 0.2
400	150 \pm 10	6-7	1.00-1.25	70	6	60	0.6-0.8	0.40-0.70	200	\pm 0.2
500	160 \pm 10	6-7	1.25-1.50	70	6	60	0.6-0.8	0.40-0.70	200	\pm 0.2

* Closer volumetric standardization must be carried out by user with substances under actual conditions of use.

understood that the vendor marks the pipets on the basis of a similar method employing an adjusted volume of mercury.

Additional reports will be made on other microvolumetric apparatus.

LITERATURE CITED

- (1) *Chem. Eng. News* 26, 883 (1948).
- (2) Committee on Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society, *ANAL. CHEM.* 21, 1283, 1555 (1949); 22, 1228 (1950); 23, 523, 1689 (1951); 26, 1186 (1954); 28, 112, 1993 (1956).

- (3) Upson, U. L., Hanford Works, General Electric Co., Richland, Wash., private communication.

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Semimicrodetermination of Bromides Application to Physiological Fluids

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► A semimicroanalytical method for bromides, based on the van der Meulen method, is described. Bromide can be evaluated in 0.01-mg. quantities (0.01 mg. of bromide ion) with an error of $\pm 3\%$. This method can be applied to the determination of bromide in physiological fluids and pre-ashing of the sample can be avoided.

LACK of a convenient and accurate microanalytical method has made it difficult to investigate the bromide

content of plant and animal fluids, such as blood, urine, and sweat, when only small quantities of these substances are available. Several publications exist on bromide distribution in living matter, but few deal with normal diet conditions; most are concerned with bromide distribution in the body after bromide administration. The methods in use are inconvenient for the determination of the bromide present under normal conditions. Procedures recommended by certain authors are

rejected by others (5, 8-10) and in a critical survey of methods up to 1936, Neufeld (6) states that only some are reliable. When bromide is determined in living matter, an initial destruction of the organic material is necessary. This is usually accomplished by an alkaline ashing process, even if the bromide ion content is of interest, and not the organically bound bromide; also the micromethod recommended by Hunter (1, 2) provides for an initial alkaline ashing treatment.